



Standard Test Method for Determination of Total Sulfur in Iron Ores and Related Materials by Combustion-Iodate Titration¹

This standard is issued under the fixed designation E 395; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of total sulfur in iron ores, concentrates, agglomerates, and related materials in the concentration range from 0.007 to 0.50 %.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁴

E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials⁵

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁵

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method⁵

3. Summary of Test Method

3.1 The sulfur in the test sample is converted to sulfur dioxide by combustion in a stream of oxygen. The sulfur dioxide is absorbed in an acidified starch-iodide solution and titrated with potassium iodate solution. The potassium iodate solution is standardized against a similar type ore of known sulfur content since the percentage of sulfur evolved as sulfur dioxide varies with different materials.

4. Significance and Use

4.1 This test method is intended to be used for compliance with compositional specifications for sulfur content. It is assumed that all who use these procedures will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed, such as those described in Guide E 882.

5. Interferences

5.1 Halogens interfere with this test method.

6. Apparatus

6.1 *Induction Heating Apparatus* for determination of sulfur by direct combustion as described in Practices E 50, Apparatus No. 13.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7.3 *Copper (Low-Sulfur) Accelerator*, chips or rings.

7.4 *Hydrochloric Acid (3+97)*—Add 3 volumes of concentrated hydrochloric acid (HCl) (sp gr 1.19) with 97 volumes of water.

7.5 *Iron (Low-Sulfur) Accelerator*, chips or powder.

7.6 *Potassium Iodate, Standard Solution* (1 mL = 0.1 mg S)—Dissolve 0.2225 g of potassium iodate (KIO₃) in 900 mL of water and dilute to 1 L.

¹ This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrated, and Related Metallurgical Materials.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ *Annual Book of ASTM Standards*, Vol 03.06.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

NOTE 1—The sulfur equivalent is based on the complete conversion of sulfur to sulfur dioxide. The recovery of sulfur as the dioxide is less than 100 %, but is consistent when the temperature and the rate of oxygen flow are maintained constant. An empirical factor must be determined by an analysis of a standard sample.

7.7 *Starch-Iodide Solution*—Transfer 1 g of soluble or arrowroot starch to a small beaker, add 2 mL of water, and stir until a smooth paste is obtained. Pour the mixture into 50 mL of boiling water. Cool, add 1.5 g of potassium iodide (KI), stir until dissolved, and dilute to 100 mL.

NOTE 2—This solution shall be prepared daily.

7.8 *Tin (Low-Sulfur) Accelerator*, granules.

8. Sampling and Sample Preparation

8.1 The gross sample shall be collected and prepared in accordance with Practice E 877.

8.2 The laboratory sample shall be pulverized to pass a No. 100 (150- μ m) sieve.

8.3 Weigh to ± 25 mg an amount of prepared sample specified as follows:

Content of Sulfur, %	Weight of Sample, g
0.005 to 0.025	2.0
0.020 to 0.10	1.0
0.10 to 0.25	0.5
0.25 to 0.50	0.2

9. Procedure

9.1 Transfer the test sample to a small, dry weighing bottle and place in a drying oven. After drying at 105 to 110°C for 1 h, cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize pressure and weigh the capped bottle to the nearest 0.001 g. Repeat the drying and weighing until there is no further weight loss. Transfer the test sample to a pre-ignited crucible and reweigh the capped bottle to the nearest 0.001 g. The difference between the two weights is the weight of the test sample.

9.2 To the crucible add 1 g of iron, 0.9 g of tin, and 1 copper ring or 0.6 g of copper chips and cover.

9.3 Select a standard sample ore, similar in type and sulfur content to the test sample. Weigh, dry, transfer to a pre-ignited crucible, and add the accelerators as in 9.1 and 9.2.

9.4 Turn on the power of the induction furnace and allow the unit to warm up. With oxygen bubbling through the absorption vessel, fill it to a pre-determined point with HCl (3+97) (Note 3). Add 2 mL of starch solution to the vessel. With the oxygen flow adjusted to 1.0 to 1.5 L/min (Note 4), add KIO₃ solution until the intensity of the blue color is that which is to be taken as the end-point. Refill the buret.

NOTE 3—Always fill the titration vessel to the same point.

NOTE 4—The oxygen flow rate may be adjusted to meet the requirements of individual operators or equipment; however, the flow rate must be the same for the test samples and the standard samples.

9.5 After the unit has warmed up for at least 45 s, place the covered crucible containing the standard sample and accelerators on the pedestal. With the oxygen flow adjusted, raise the crucible, close the furnace, and turn on the power. Burn the sample for 8 to 10 min. Titrate continuously with the KIO₃ solution at such a rate as to maintain as nearly as possible the original intensity of the blue color. The end point is reached

when the original blue color is stable for 1 min without further addition of KIO₃ solution. Record the final buret reading and drain the titration vessel through the exhaust stopcock.

NOTE 5—If the blue color in the cell disappears or white fumes escape the surface of the cell, low bias may result.

9.6 Refill the titration vessel with the HCl (3+97), add 2 mL of the starch solution, and titrate with the KIO₃ solution to the pre-selected end-point color. Refill the buret, place the covered crucible containing the test sample and accelerators on the pedestal of the furnace, and proceed as in 9.5.

10. Blank

10.1 Determine the blank by placing the same amount of accelerators used in the test sample in a pre-ignited crucible. Cover and proceed as in 9.5.

11. Calculation

11.1 Calculate the sulfur factor of the potassium iodate as follows:

$$\text{Sulfur factor, g/unit volume} = (A \times B) / [(C - D) \times 100] \quad (1)$$

where:

A = standard sample used, g,

B = sulfur in the standard sample, %,

C = KIO₃ solution required for titration of the standard sample (Note 6), mL, and

D = KIO₃ solution required for titration of the blank (Note 6) mL.

NOTE 6—Or apparent percentage of sulfur for “direct-reading” burets.

11.2 Calculate the percentage of sulfur in the test sample as follows:

$$\text{Sulfur, \%} = [(E - D)F/G] \times 100 \quad (2)$$

where:

E = KIO₃ solution required for titration of the test sample (Note 5),

D = millilitres of KIO₃ solution required for titration of the blank,

F = sulfur factor of the KIO₃ solution in g/unit volume, and

G = grams of test sample used.

12. Report

12.1 Round the test results that are less than 0.01 % to the nearest 0.001 % in accordance with Practice E 29.

12.2 Report results below the lower scope limit as approximate, for example: ~ 0.000 , ~ 0.001 , ... ~ 0.006 .

12.3 Round test results in the range of 0.01 to 0.50 % to the nearest 0.01 % in accordance to Practice E 29.

12.4 Report results greater than 0.5 % as >0.5 %.

13. Precision and Bias

13.1 *Precision*—Nine laboratories cooperated in obtaining the data summarized in Table 1.

13.2 *Bias*—No information on the bias of this test method is known. Accepted reference materials were not included in the materials used in the interlaboratory study. Users of the test

TABLE 1 Statistical Information

Average Sulfur Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
0.004	0.0016	0.0033
0.054	0.0033	0.0147
0.15	0.010	0.022
0.25	0.012	0.029
0.45	0.023	0.024

difference between the accepted value for the sulfur concentration and the mean value from interlaboratory testing of the reference material.

14. Keywords

14.1 agglomerates; concentrates; iron ores; sulfur content

method are encouraged to employ accepted reference materials, if available, and to judge the bias of the method from the

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